भारतीय मानक
Indian Standard

IS 17983 : 2022

3,4 — डाइक्लोरोफिनाइल आइसोसाइनेट — विशिष्टि

3,4 — Dichlorophenyl Isocyanate — Specification

ICS 71.080.99

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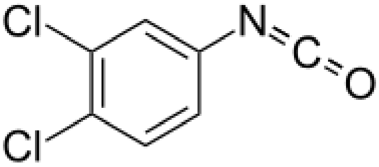
October 2022 **Price Group 7**

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 9

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

3,4-Dichlorophenyl Isocyanate is an organic compound with the formula $C_7H_3Cl_2NO$. It is used as an intermediate in organic synthesis. It is irritant to many tissues, including eyes and mucous membranes, and inhaling dust from the chemical may result in respiratory harm. It is represented by the following structural formula:



Molecular mass: 188.01 $g.mole^{-1}$
CAS registry number: 102-36-3

Based on the recommendations of Department of Chemicals and Petrochemicals, Ministry of Chemicals and Fertilizer, the Committee has decided to formulate Indian Standard on 3,4-dichlorophenyl isocyanate.

Safety instructions to be followed during testing are given in informative Annex D

The composition of the Committee, responsible for the formulation of this standard is given at Annex E.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

3,4-DICHLOROPHENYL ISOCYANATE — SPECIFICATION

1 SCOPE

This standard prescribes the requirements, methods of sampling and testing for 3,4-dichlorophenyl isocyanate.

2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

IS No.	*Title*
5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)
5762 : 1970	Method for determination of melting point and melting range

3 REQUIREMENTS

3.1 Description

The material shall be a white to pale yellow solid at room temperature (25 ± 2)°C. It is having a low melting point of 41°C.

3.2 The material shall also comply with the requirements given in Table 1 when tested as prescribed in col 4 and col 5 of Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable drums, tankers, ISO containers etc, as agreed between purchaser and supplier.

4.2 Marking

4.2.1 Each container shall be securely closed and shall bear the following information:

a) Name of the material;
d) Name of manufacturer and his recognized trade-mark, if any;
c) Month and year of manufacture;
d) Lot or batch number;
e) Net weight and gross weight;
f) Label: Acute Toxic, Irritant and Health Hazard; and
g) Any other statutory requirement.

4.2.2 Each container shall bear the minimum cautionary notice worded as under:

TOXIC

AVOID CONTACT WITH SKIN

KEEP WELL CLOSED AND PROTECTED FROM LIGHT

4.2.3 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed there under, and the products may be marked with the Standard Mark.

5 SAMPLING

5.1 The method of drawing representative samples of the material shall be as prescribed in **4** of IS 5299.

5.2 Criteria for Conformity

For declaring the conformity of a lot to the requirements of all the other characteristics tested on the composite sample, the test result for each of the characteristics shall satisfy the relevant requirement given in Table 1.

6 TEST METHODS

6.1 Test shall be conducted as prescribed in col 4 and col 5 of Table 1.

6.2 Unless specified otherwise, ‘pure chemicals’ and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities, which affect the result of analysis.

Table 1 Requirements for 3,4-Dichlorophenyl Isocyanate
(*Clause* 3.2, 5.2 *and* 6.1)

Sl No.	**Characteristic**	**Requirement**	**Methods of Test, Refer to**	
			Annex	IS
(1)	(2)	(3)	(4)	(5)
i)	Purity/Assay, percent by mass, *Min*	98.0	A	—
ii)	Melting point[1], °C	41 – 43	—	5762
iii)	Other Isocyanate (*as* unknown peaks) by GC, percent by mass, *Max*	0.8	A	—
iv)	Organic solvent (ortho-dichloro-benzene, tri-chlorobenzene) by GC, percent by mass, *Max*	0.8	A	—
v)	Hydrolyzable chlorine, percent by mass, *Max*	0.3	B	—
vi)	Acidity (*as* HCl), percent by mass, *Max*	0.1	C	—

[1] Sl No. (ii), Melting point requirement is optional.

ANNEX A

[*Table* 1, *Sl No.* (i), (iii) *and* (iv)]

DETERMINATION OF 3,4-DICHLORORPHENYL ISOCYANATE (PURITY/ASSAY), OTHER ISOCYANATE AND SOLVENT BY GAS CHROMATOGRAPHY

A-1 GENERAL

The test method is related to determine the purity of 3,4-dichlororphenyl isocyanate by gas chromatography and in addition provides a means for identification of certain known impurities. A sample of 3,4-dichlorophenyl isocyanate is injected on a gas chromatographic capillary column and the results are calculated using the external standard method with area normalization.

A-2 APPARATUS

A-2.1 Gas Chromatograph

A-2.1.1 Any gas chromatograph equipped with a flame ionization detector (FID), a split-splitless injector and a suitable electronic integrator/software can be used with following accessories and operating condition:

Column	:	Capillary column of fused silica, with (5 percent phenyl)-methylpolysiloxane phase, with length 30 m; internal diameter 0.32 mm; and film thickness 0.32 µm.
Pressure at head of column	:	151.685 kPa (22 psi)
Carrier gas	:	Hydrogen/Nitrogen/Helium
Fuel gas and flow rate	:	Hydrogen: 40 ml/min; Air : 400 ml/min
Make up flow	:	As per the requirement of instrument make/model.
Sample preparation	:	20 percent solution in toluene
Injection volume	:	1 µl
Split flow	:	60 ml /min
Detector	:	FID

A-2.1.2 *Temperature Programme of Oven, Detector and Injector*

Injector Temperature, °C	*Detector Temperature, °C*	*Oven*		
		Temperature, °C	*Hold Time, min*	*Ramp Rate, °C / min*
250	250	125	2	6
		210	1	20
		240	10	—

NOTE — The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packed/capillary, diameter, length, film thickness etc), calibration technique (internal standard, external standard, area normalization, percent area etc.), carrier gas (He, H_2, N_2) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.

A-2.2 Syringe, 10 µl

A-2.3 Analytical Balance, with Least Count of 0.1 mg

A-3 REAGENT

A-3.1 Certified Reference Material (CRM) of 3, 4-Dichlorophenyl Isocyanate.

A-3.2 *m*-phenylene diisocyanate.

A-3.3 1,2,4-Trichlorobenzene and 1,2,3-Trichlorobenzene.

A-3.4 *o*-**Dichlorobenzene**.

A-3.5 Toluene

A-4 SAMPLING

Take a clean and dry 500 ml glass bottle. Do not expose the sample to moisture. Moisture reacts with 3,4-dichlorophenyl isocyanate to form solids.

A-5 CALIBRATION

A-5.1 Prepare a standard solution containing the components given below (*see* **A-3.1** to **A-3.5**), of the specified concentration. A typical example of standard solution is given below for reference.

Component	*Weight, g*	*Concentration in percent, (w/w)*
3,4-dichlorophenyl isocyanate	80.000 0	98.401 0
o-dichlorobenzene	1.000 0	1.230 0
m-phenylene diisocyanate	0.100 0	0.123 0
1,2,4-trichlorobenz-ene	0.100 0	0.123 0
1,2,3-trichlorobenz-ene	0.100 0	0.123 0

A-5.2 Take 2 g calibration blend into 10 ml volumetric flask and makeup with toluene (that is 20 percent solution in toluene).

A-5.3 Make sure the GC instrument is adjusted to the conditions stated as above. Inject 1.0 µl of the standard at least three times and obtain the chromatograms. Calculate the calibration correction factor for each component except toluene (solvent use for sample preparation) by dividing its concentration by the respective area in its chromatogram. Find the arithmetic mean of the factors calculated for each component. Each factor is calculated to 4 decimal places. When using the factors, the calculation of the concentration of the sample is done using the mean obtained or use suitable software for auto calibration with respect to mass of calibration standard.

A-6 SAMPLE PROCDURE

Take 2 g sample into 10 ml volumetric flask and make it up with toluene (that is 20 percent solution in toluene). Inject 1.0 µl of the sample into the chromatograph.

A-7 CALCULATION

A-7.1 Sum the areas of all unknown components.

A-7.2 Multiply the area of each component of the sample except toluene (solvent use for sample preparation) by its relative response factor as mentioned in **A-5**. Obtain the sum of all corrected areas. Normalize each corrected area to 100 percent and report 3,4-dichlorophenyl isocyanate, ortho-dichlorobenzene and trichlorobenzene as solvent and Other Isocyanate as unknown compounds or use suitable software for auto calculation with respect to calibration standard.

NOTE — Use response correction factor = 1, for the unknown components.

ANNEX B

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF HYDROLYZABLE CHLORINE

B-1 GENERAL

This method is used for the determination of hydrolyzable chlorine content of 3,4-dichlorophenyl isocyanate. The method is applicable to the determination of hydrolyzable chlorine content in the range of 0 to 0.50 percent.

B-2 PRINCIPLE

The isocyanate containing hydrogen chloride, carbonyl dichloride (CDC), carbamyl chlorides, aromatic acid chlorides and chloromethyl compounds is reacted with an aqueous methanol solution to convert the organic chloro compounds to free hydrochloric acid. The liberated HCl is then titrated with silver nitrate solution, potentiometrically.

B-3 SAMPLING

Take a clean and dry 500 ml glass bottle. Do not expose the sample to moisture. Moisture reacts with toluene diisocyanate to form solids.

B-4 APPARATUS

B-4.1 Magnetic stirrer

B-4.2 Titrator

with a silver electrode versus an Ag/AgCl reference electrode or combine silver ring electrode.

B-4.3 Analytical Balance

B-4.4 Hotplate

B-5 REAGENTS

B-5.1 Methanol

B-5.2 Silver Nitrate ($AgNO_3$), 0.014 1 N standard

Dissolve 2.395 g silver nitrate in distilled water and make up to 1.0 l.

B-5.3 1:1 Nitric acid

Take 1 volume of 70 percent nitric acid (HNO_3) and 1 volume of distilled water.

B-6 PROCEDURE

Weigh 13.5 ± 1.5 g of sample using any glass sampling device into a clean, dry 400 ml beaker. Add 125 ml methanol using graduated measuring cylinder and agitate on the magnetic stirrer. When the reaction starts, which is usually within a few seconds, slowly add 100 to 125 ml of chloride free distilled water. Cover with a clean watch glass and agitate for 10 to 15 min. Transfer the beaker from the stirrer to a hot plate to digest the content at approximately 65 to 70°C for about 30 min. Wash down the sides of the beaker with distilled water and cool to room temperature by means of an ice bath. Add 10 ml of 1 : 1 nitric acid to the sample. Titrate the sample with 0.014 1 N silver nitrate using automatic titrator. From the instrument display, determine the volume of the solution used. Run blanks exactly in a same manner without adding sample.

B-7 CALCULATION

$$\text{Hydrolyzable chlorine, percent} = \frac{(A-B) \text{ x } N \text{ x } 3.546}{S}$$

where

A = volume silver nitrate ($AgNO_3$), for sample titration, ml;

B = volume silver nitrate ($AgNO_3$) for blank titration, ml;

N = normality silver nitrate ($AgNO_3$) solution; and

S = sample weight, g

ANNEX C

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF ACIDITY

C-1 GENERAL

The isocyanates are reacted with excess *n*-propanol to form urethanes. The acidic components released are absorbed in the reaction mixture and titrated potentiometrically with methanolic potassium hydroxide.

C-2 SAMPLING

Take a clean and dry 500 ml glass bottle. Do not expose the sample to moisture. Moisture reacts with toluene diisocyanate to form solids.

C-3 APPARATUS

C-3.1 Automatic titrator, complete with reference and glass electrode for *p*H measurement.

C-3.2 Buffer Solutions, *p*H 4 and 7.

C-3.3 Beaker, 250 ml.

C-4 REAGENTS

C-4.1 Potassium Hydroxide, 0.01 N methanolic, standardized to ± 0.000 1 N

Take 0.66 g potassium hydroxide (assay 85 percent) in dry 1.0 l volumetric flask and dilute it up to mark with methanol. Stir thoroughly for 15 min.

C-4.2 Hydrochloric acid, 0.01 N, standardized

Take 1.042 g hydrochloric acid (assay 35 percent) in dry 1.0 l volumetric flask and dilute it up to mark with methanol. Stir thoroughly for 15 min.

C-4.3 *n*-Propanol.

C-5 PROCEDURE

Prepare the automatic titrator according to its operation manual. If necessary, standardize instrument to *p*H 4 with buffer solution. Take 100 ml of *n*-propanol in a 250 ml glass beaker and dip the combined glass electrodes in *n*-propanol. Adjust its *p*H to 5.0 with either 0.01 N alcoholic potassium hydroxide or 0.01 N hydrochloric acid carefully. Remove electrodes from the solution. Add 10 ± 0.1 gm of the sample into beaker while agitating the content. Record the weight of sample. Cover with a watch glass and continue agitation for 10 min. Immerse electrodes in sample solution and titrate with 0.01 N alcoholic potassium hydroxide, till the endpoint is reached. The equivalence point will occur in the range of about 5.50 to 7.00 *p*H units (end point should be a greatest equivalence point between *p*H 5.5 to 7.00).

C-6 CALCULATION

Acidity (*as* HCl), percent =

$$\frac{A \text{ x } N \text{ x } 3.647}{S}$$

where

A = volume of potassium hydroxide solution required, ml;

N = normality of potassium hydroxide solution; and

S = mass of sample, g

C-7 REPORT

Report results to nearest 0.001 percent.

ANNEX D

(*Informative Annex*)

D-1 SAFETY INSTRUCTIONS TO BE TAKEN DURING TESTING

D-1.1 General Safety Instructions

a) Avoid skin contact and do not inhale fumes.

b) Use adequate ventilation to keep the vapor concentration at a minimum.

c) Any spills of nitric acid must be washed off immediately with cold water. Use rubber gloves, an apron and goggles when handling these reagents.

d) Methanol is flammable. Keep away from sparks and flames

ANNEX E

(*Foreword*)

COMMITTEE COMPOSITION

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

Organization	*Representative(s)*
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